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(54) Title: METHOD FOR MANUFACTURING COBALT CATALYSTS

(57) Abstract

A method for manufacturing cobalt complexes having the formula: $[Co(NH_3)_5M]\ T_y$ wherein M ligands are selected from substituted and unsubstituted C_1-C_{30} carboxylic acids having the formula: RC(O)O-; said method comprising reacting cobalt complexes having the formula: $[Co(NH_3)_5X]\ T_y$ (e.g., X is chlorine) with concentrated ammonium hydroxide followed by carboxylic acid anhydride of the formula: RC(O)O(O)CR.

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METHOD FOR MANUFACTURING COBALT CATALYSTS

TECHNICAL FIELD

The present invention relates to methods for manufacturing cobalt complexes having the formula:

$$[Co(NH_3)_5M]\ T_y$$

wherein M ligand is selected from substituted and unsubstituted C_1-C_{30} carboxylic acids having the formula:

$$RC(O)O\text{-}.$$

These catalysts are particularly useful in bleach-containing consumer compositions, especially automatic dishwashing detergents and laundry detergents comprising bleach.

BACKGROUND OF THE INVENTION

Cobalt catalysts are well known, as are a variety of methods for manufacturing them. Most synthesis methods, however, are directed simply to methods effective for obtaining experimental quantities for academic studies. These are described, for example, in M. L. Tobe, "Base Hydrolysis of Transition-Metal Complexes", Adv. Inorg. Bioinorg. Mech., (1983), 2, pages 1-94; J. Chem. Ed. (1989), 66 (12), 1043-45; The Synthesis and Characterization of Inorganic Compounds, W.L. Jolly (Prentice-Hall; 1970), pp. 461-3; Inorg. Chem., 18, 1497-1502 (1979); Inorg. Chem., 21, 2881-2885 (1982); Inorg. Chem., 18, 2023-2025 (1979); Inorg. Synthesis, 173-176 (1960); and Journal of Physical Chemistry, 56, 22-25 (1952).

For use in consumer products, however, it is necessary that the cobalt catalysts be prepared in large quantities by the most cost effective manner with the highest possible purity. It has been discovered by the present invention that cobalt catalysts containing carboxylate ligands can be prepared on an industrially useful scale by the present process.

BACKGROUND ART

U.S. Patent 4,810,410, to Diakun et al, issued March 7,1989; U.S. 5,246,612, to Van Dijk et al., issued September 21, 1993; U.S. 5,244,594, to Favre et al., issued September 14, 1993; and European Patent Application, Publication No. 408,131, published January 16, 1991 by Unilever NV, see also: U.S. Patent 5,114,611, to Van

Kralingen et al, issued May 19, 1992 (transition metal complex of a transition metal, such as cobalt, and a non-macro-cyclic ligand); U.S. Pat. 4,430,243, to Bragg, issued February 7, 1984 (laundry bleaching compositions comprising catalytic heavy metal cations, including cobalt); German Patent Specification 2,054,019, published October 7, 1971 by Unilever N.V. (cobalt chelant catalyst); and European Patent Application Publication No. 549,271, published June 30, 1993 by Unilever PLC (macrocyclic organic ligands in cleaning compositions).

SUMMARY OF THE INVENTION

The present invention relates to methods for manufacturing cobalt complexes having the formula:

$$[Co(NH_3)_5M]\ T_y$$

wherein the M ligand is selected from substituted and unsubstituted C_1-C_{30} carboxylic acids having the formula:

$$RC(O)O\text{-};$$

said method comprising the steps of:

(a) reacting a cobalt (III) pentaamine complex having the formula:

$$[Co(NH_3)_5X]\ T_y$$

wherein X is selected from base labile ligands (preferably Cl^-, Br^-, OH^-, H_2O, NO_2^-, SO_4^{2-}, and CO_3^{2-}); and T is one or more counteranions present in a number y, where y is an integer to obtain a charge-balanced salt (preferably y is 1 or 2);

with an aqueous base (preferably concentrated ammonium hydroxide); followed by

(b) reacting the product of step (a) with a carboxylic acid anhydride of the formula:

$$RC(O)O(O)CR$$

wherein each R is independently selected from substituted or unsubstituted C_1-C_{30} moieties (preferably both R are the same); and

(c) collecting the cobalt complex.

All parts, percentages and ratios used herein are expressed as percent weight unless otherwise specified. All documents cited are, in relevant part, incorporated herein by reference.

DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to a method for manufacturing cobalt complexes having the formula:

$[Co(NH_3)_5M]\ T_y$

wherein the M ligands are selected from substituted and unsubstituted C_1-C_{30} carboxylic acids having the formulas:

RC(O)O-.

This method comprises the first step of reacting with an aqueous base a cobalt (III) pentaamine complex having the formula:

$[Co(NH_3)_5X]\ T_y$

wherein X is selected from base labile ligands. Preferred X are selected from Cl^-, Br^-, OH^-, H_2O, NO_2^-, SO_4^{2-}, and CO_3^{2-}. T is one or more appropriately selected counteranions present in a number y, where y is an integer to obtain a charge-balanced salt (preferably y is 1 or 2). Preferred T are selected from the group consisting of chloride, nitrate, nitrite, sulfate, citrate, acetate, carbonate, bromide, PF_6^-, BF_4^-, phosphate, tosylate, methanesulfonate, and combinations thereof.

The aqueous base for this first step is preferably concentrated aqueous ammonium hydroxide (preferably at least 20%, more preferably at least 25%, and typically between 28-32% solutions). Other aqueous bases include, for example, potassium hydroxide, sodium hydroxide, and other weak amine bases.

Reaction conditions for this step typically require slight heating (above about 65°C) sufficient to dissolve the cobalt (III) pentaamine complex, followed by cooling to approximately room temperature prior to the next step.

This first step is followed by reacting the product of the first step with a carboxylic acid anhydride of the formula:

RC(O)O(O)CR

wherein each R is independently selected from substituted or unsubstituted C_1-C_{30} moieties. It is preferred that both R moieties are the same, but mixed anhydrides may be used as desired for the desired cobalt complex being synthesized. Reaction conditions for this step are typically room temperature for from about 10 to about 60 minutes.

At the end of the reaction process, the cobalt complex is collected. Preferred collection methods include, for example, evaporation to remove the solvent or lyophilization. The cobalt complex collected may be used as is, or further purified or modified for incorporation into the desired product or use to be made of the complex. This includes optionally washing the solid product with ethanol to remove non-cobalt salts.

The present invention method preferably is carried out in one reaction vessel without isolation or separation of the intermediate reaction products. However, if desired, one or more of the reaction steps may be conducted in separate reaction vessels, and may be followed or preceeded by optional separation and/or collection steps of the intermediate reaction materials.

Herein, R is preferably selected from the group consisting of hydrogen and C_1-C_{30} (preferably C_1-C_{18}) unsubstituted and substituted alkyl, C_6-C_{30} (preferably C_6-C_{18}) unsubstituted and substituted aryl, and C_3-C_{30} (preferably C_5-C_{18}) unsubstituted and substituted heteroaryl, wherein substituents are selected from the group consisting of $-NR'_3$, $-NR'_4^+$, -C(O)OR', -OR', $-C(O)NR'_2$, wherein R' is selected from the group consisting of hydrogen and C_1-C_6 moieties. Such substituted R therefore include the moieties $-(CH_2)_nOH$ and $-(CH_2)_nNR'_4^+$, wherein n is an integer from 1 to about 16, preferably from about 2 to about 10, and most preferably from about 2 to about 5.

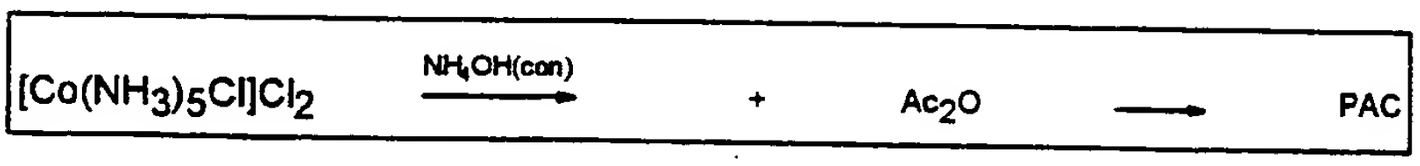
Most preferred M are carboxylic acids having the formula above wherein R is selected from the group consisting of hydrogen, methyl, ethyl, propyl, straight or branched C_4-C_{12} alkyl, and benzyl. Most preferred R is methyl. Preferred carboxylic acid M moieties include formic, benzoic, octanoic, nonanoic, decanoic, dodecanoic, and especially acetic acid. Therefore, most preferred methods use the anhydrides comprising these carboxylic acids, and preferred anhydrides are the same acids. Most preferred anhydride is acetic anhydride.

The most preferred cobalt complex prepared by the present invention is cobalt pentaamine acetate chloride, $[Co(NH_3)_5OAc]Cl_2$ (herein "PAC").

The starting cobalt (III) pentaamine complexes useful herein, the $[Co(NH_3)_5X]$ T_y complexes, are commercially available and can be prepared by a variety of methods. These include the preferred starting material $[Co(NH_3)_5Cl]Cl_2$, sold by Aldrich and by Pfaltz and Bauer.

The following nonlimiting example further illustrates the method according to the present invention.

Synthesis of $[Co(NH_3)_5OAc]Cl_2$



$[Co(NH_3)_5Cl]Cl_2$ (26.4 g, 0.10 mol) is added to distilled water (800 mL). NH_4OH (23.4 mL, 0.600 mol) is slowly added with stirring. The solution is then heated to 75°C and the solid dissolves with stirring. The solution is cooled to RT. Acetic anhydride (30.6 g, 0.30 mol) is slowly added with stirring. The solution is stirred 1 hour at RT. At this point the reaction solution can either be lyophilized to a pink powder or the solution can be rotovapped down and the resulting solid pumped on overnight at 0.05 mm. to remove residual water and NH_4OAc. The excess ammonium acetate and ammonium chloride salts can also be removed by washing the solid with ethanol. Yield 35 gr., 78.1% by uv-vis spectroscopy. HPLC [according to the method of D.A. Buckingham, et al, Inorg. Chem., 28, 4567-4574 (1989)] shows all of the cobalt is present as $[Co(NH_3)_5OAc]Cl_2$.

What is Claimed is:

1. A method for manufacturing cobalt complexes having the formula:

$$[Co(NH_3)_5M]\ T_y$$

wherein the M ligand is selected from substituted and unsubstituted C_1-C_{30} carboxylic acids having the formula:

$$RC(O)O\text{-};$$

wherein R is selected from substituted or unsubstituted C_1-C_{30} moieties;

said method comprising the steps of:

(a) reacting a cobalt (III) pentaamine complex having the formula:

$$[Co(NH_3)_5X]\ T_y$$

wherein X is selected from base labile ligands; and T is one or more counteranions present in a number y, where y is an integer to obtain a charge-balanced salt;

with an aqueous base; followed by

(b) reacting the product of step (a) with a carboxylic acid anhydride of the formula:

$$RC(O)O(O)CR$$

wherein each R is independently selected from substituted or unsubstituted C_1-C_{30} moieties; and

(c) collecting the cobalt complex.

2. The method according to Claim 1 wherein X is selected from Cl^-, Br^-, OH^-, H_2O, NO_2^-, SO_4^{2-}, and CO_3^{2-}.

3. The method according to either of Claims 1 or 2 wherein the aqueous base is ammonium hydroxide.

4. The method according to any of Claims 1-3 wherein each R in the anhydride is independently selected from the group consisting of hydrogen and C_1-C_{30} unsubstituted and substituted alkyl, C_6-C_{30} unsubstituted and substituted aryl, and C_3-C_{30} unsubstituted and substituted heteroaryl, wherein substituents are selected from the group consisting of $-NR'_3$, $-NR'_4^+$, -C(O)OR', -OR', $-C(O)NR'_2$, wherein R' is selected from the group consisting of hydrogen and C_1-C_6 moieties.

5. The method according to any of Claims 1-4 wherein each R in the anhydride is independently selected from C_1-C_{18} unsubstituted and substituted alkyl.

6. The method according to any of Claims 1-5 wherein the M ligand is a carboxylic acid moiety selected from formic, benzoic, octanoic, nonanoic, decanoic, dodecanoic, and preferably acetic acid.

7. A method for manufacturing cobalt complexes having the formula:

$$[Co(NH_3)_5M]\ T_y$$

wherein the M ligand is selected from substituted and unsubstituted C_1-C_{30} carboxylic acids having the formula:

$$RC(O)O\text{-};$$

wherein R is selected from C_1-C_{18} unsubstituted and substituted alkyl moieties;

said method comprising the steps of:

(a) reacting a cobalt (III) pentaamine complex having the formula:

$$[Co(NH_3)_5X]\ T_y$$

wherein X is selected from Cl^-, Br^-, OH^-, H_2O, NO_2^-, SO_4^{2-}, and CO_3^{2-}; and T is one or more counteranions present in a number y, where y is an integer to obtain a charge-balanced salt;

with concentrated ammonium hydroxide; followed by

(b) reacting the product of step (a) with a carboxylic acid anhydride of the formula:

$$RC(O)O(O)CR$$

wherein each R is independently selected from C_1-C_{18} unsubstituted and substituted alkyl moieties; and

(c) collecting the cobalt complex.

8. The method according to any of Claims 1-7 wherein both R in the anhydride are the same moiety selected from the group consisting of hydrogen, methyl, ethyl, propyl, straight or branched C_4-C_{12} alkyl, and benzyl.

9. The method according to any of Claims 1-8 wherein the anhydride is acetic anhydride.

10. The method according to any of Claims 1-5 and 7 wherein each R in the anhydride is independently selected from the moieties $-(CH_2)_nOH$ and $-(CH_2)_nNR'_4^+$, wherein n is an integer from 1 to 16, preferably from 2 to 10.

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(30) Priority Data:
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(71) Applicant: THE PROCTER & GAMBLE COMPANY [US/US]; One Procter & Gamble Plaza, Cincinnati, OH 45202 (US).

(72) Inventor: PERKINS, Christopher, Mark; 7230 Fernbank Avenue, Cincinnati, OH 45233 (US).

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(54) Title: METHOD FOR MANUFACTURING COBALT CATALYSTS

(57) Abstract

A method for manufacturing cobalt complexes having the formula: $[Co(NH_3)_5M]\ T_y$ wherein M ligands are selected from substituted and unsubstituted C_1-C_{30} carboxylic acids having the formula: RC(O)O-; said method comprising reacting cobalt complexes having the formula: $[Co(NH_3)_5X]\ T_y$ (e.g., X is chlorine) with concentrated ammonium hydroxide followed by carboxylic acid anhydride of the formula: RC(O)O(O)CR.

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INTERNATIONAL SEARCH REPORT

International Application No
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A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C07F15/06 C11D3/39

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C07F C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	INORGANIC CHEMISTRY, vol. 18, no. 6, 1979, EASTON US, pages 1497-1502, XP002018152 L.M. JACKMAN: "Synthesis of Transition-Metal Carboxylato Complexes" cited in the application see particularly page 1498, right-hand column, paragraph (ii) ---	1-10
A	GB 3 034 061 A (FUJI PHOTO FILM CO.) 29 May 1980 see particularly page 6, synthesis example 4 --- -/--	1-10

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
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Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl, Fax (+ 31-70) 340-3016	Authorized officer Beslier, L

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	INORGANIC CHEMISTRY, vol. 21, no. 7, 1982, EASTON US, pages 2881-2885, XP002018153 THOMAS J. WIERENGA: "Synthesis and Characterization of Cobalt(III) Nicotinic Acid Complexes" cited in the application see the whole document -----	1-10

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/US 96/08662

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
GB 3034061 A		NONE	

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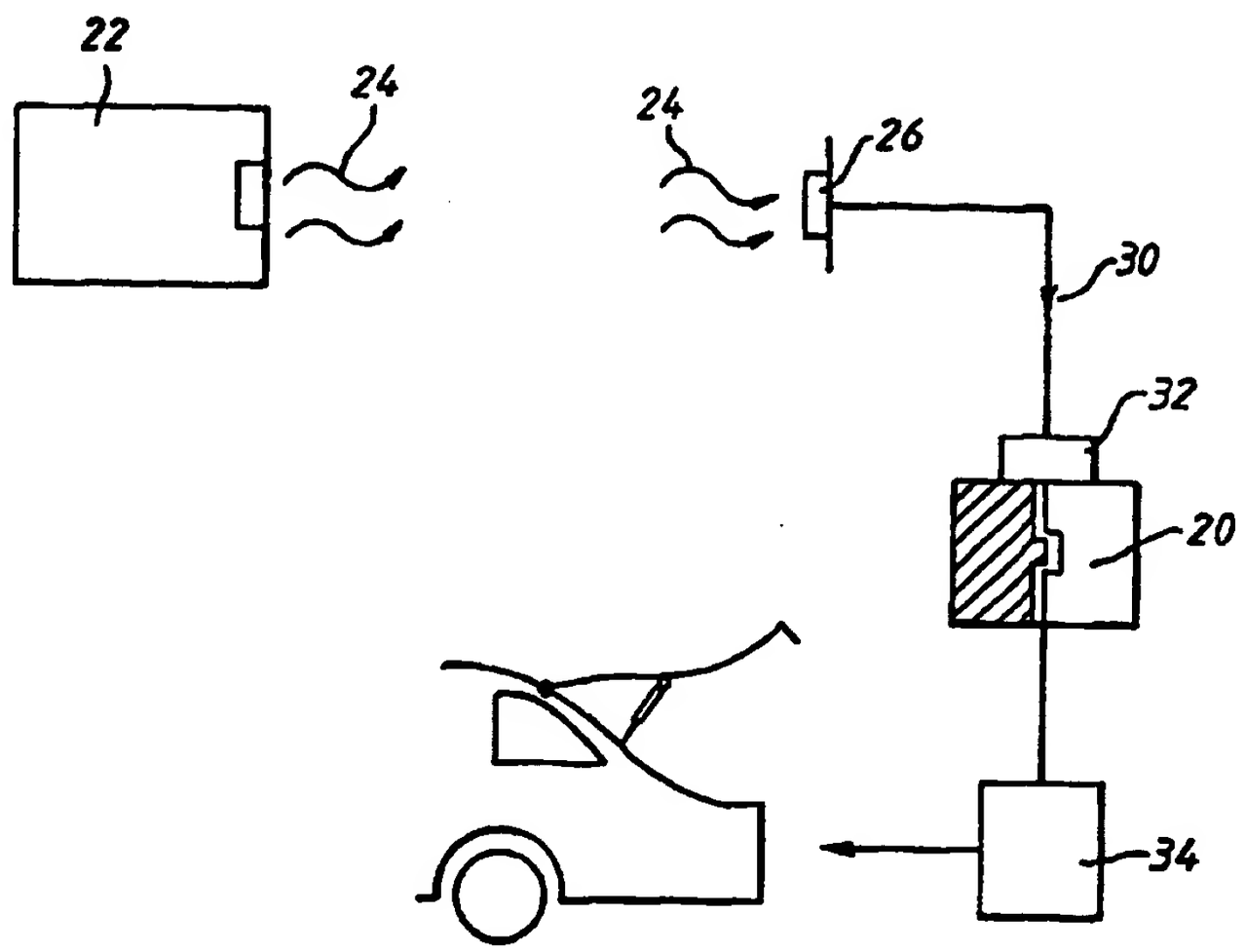
(54) Title: OPENING ARRANGEMENTS AND METHODS FOR CLOSURE MEMBERS



(57) Abstract

An arrangement and method for remotely unlocking and raising the hatchback (12) of a motor vehicle (10) is described. Firstly, a signal (24) is sent to unlock and unlatch the vehicle hatchback (12). The unlatched hatchback (12) is automatically raised by an amount sufficient to allow gas springs (18) to complete the raising operation. The gas springs (18) may incorporate a device to prevent the hatchback being fully raised.

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ES	Spain	**MG**	Madagascar	**UG**	Uganda
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OPENING ARRANGEMENTS AND METHODS FOR CLOSURE MEMBERS

The invention relates to a closure opening method, comprising the steps of emitting a signal from a remote location; detecting the signal; and causing an unlatching mechanism to be operated.

The invention further relates to a remotely operable closure opening arrangement, comprising a releasable latching mechanism for latching the closure in the closed position, signal emitting means for emitting an opening signal from a remote location, and detecting means for detecting the signal and releasing the latching mechanism.

Such arrangements and methods are shown in US 4,796,932. In this system, for remotely releasing a motor vehicle compartment panel, a remote release and pull down unit is provided which allows the panel to be unlatched and pulled down enabling the user to unlatch and latch the panel remotely. However, it is desirable to produce a closure opening arrangement in which the panel is opened by a predetermined amount in a controlled fashion.

Accordingly, the known method is characterised by the steps of opening the closure to an initial extent sufficient for a controlled opening mechanism to become operative and causing the closure to be opened by the controlled opening mechanism to a predetermined greater extent.

Furthermore, the known arrangement is characterised by initial opening means responsive to release of the latching mechanism to open the closure to an initial position, and a controlled opening

mechanism operative to open the closure from the initial partially open position to a predetermined greater extent.

Hatchback and luggage compartment opening arrangements, embodying the invention, for use in motor vehicles will now be described, by way of example only, with reference to the accompanying diagrammatic drawings in which:

Figure 1 is a side view of the rear of a motor vehicle showing the hatchback in a closed position;

Figure 2 is a side view of the rear of a motor vehicle showing the hatchback in a fully raised position; and

Figure 3 is a schematic block diagram of the controls of an embodiment of the invention.

The motor vehicle 10 has a hatchback 12 pivotally attached to the body of the vehicle 14 by hinges 16. The hatchback 12 is retained in the closed position by means of a latch and a locking mechanism 20. The latch and locking mechanism 20 can be operated remotely by a portable key device 22 (see Figure 3).

Gas springs 18 (Figure 2, which shows only one of the gas springs) are provided for lifting the hatchback 12 and retaining it in the open position.

To open the hatchback, in accordance with the invention, the operator presses the appropriate button on the separate key device. A suitable signal (such as an infra-red or radio signal) is accordingly emitted by the key device 22 which in turn is

detected by a detector 26. The detector 26 emits a signal on a line 30 that activates a component 32 to unlock the latch mechanism 20.

A spring 34 or any other suitable means then raises the unlatched hatchback 12 slightly and to a level where the gas springs 18 can become operative and raise the hatchback.

The control unit 32 can be part of the central door locking system. However, preferably it can cause the hatchback to open independently of the other vehicle doors.

Additionally, the key device 22 may have other functions such as unlocking the central door locking or arming the vehicle alarm and need not be solely for raising the hatchback.

Furthermore, it will be appreciated that the vehicle need not be a hatchback motor vehicle, and the vehicle part to be opened need not be the hatchback, but could for example be another closure member which is opened by a gas spring or similar means when unlatched, such as the lid of a luggage compartment.

In a modified embodiment of the invention, the gas springs 18 allow the hatchback 12 to be raised only to a certain predetermined intermediate level less than the fully open position. This reduces the risk that the hatchback 12 will be damaged by contacting a low roof or other obstruction. The hatchback can thereafter be caused to rise to the fully open position if desired, by manual force or by further unlatching action.

CLAIMS

1. A closure opening method, comprising the steps of emitting a signal (24) from a remote location; detecting the signal (24); and causing an unlatching mechanism (20) to be operated; characterised by the steps of opening the closure (12) to an initial extent sufficient for a controlled opening mechanism (18) to become operative; and causing the closure (12) to be opened by the controlled opening mechanism (18) to a predetermined greater extent.

2. A method according to claim 1, characterised in that the predetermined extent to which the closure (12) is opened by the controlled opening mechanism (18) is less than the fullest extent of opening.

3. A method according to claim 2, characterised in that the closure (12) can be opened to the fullest extent from the predetermined extent.

4. A remotely operable closure opening arrangement, comprising a releasable latching mechanism (20) for latching the closure in the closed position, signal emitting means (22) for emitting an opening signal (24) from a remote location, and detecting means (26) for detecting the signal (24) and releasing the latching mechanism (20), characterised by initial opening means (34) responsive to release of the latching mechanism (20) to open the closure (12) to an initial position, and a controlled opening mechanism (18) operative to open the closure from the initial partially open position to a predetermined greater extent.

5. An arrangement according to claim 4, characterised in that the predetermined greater extent to which the closure (12) is opened by the controlled opening mechanism (18) is less than the fullest extent of opening.

6. An arrangement according to claim 5, characterised in that the closure (12) can be opened to the fullest extent from the predetermined extent.

7. An arrangement or method according to any preceding claim characterised in that the signal (24) is an infra-red signal.

8. An arrangement or method according to any preceding claim characterised in that the signal (24) is a radio signal.

9. An arrangement or method according to any preceding claim, characterised in that the unlatching mechanism (20) is part of a central door locking system in a motor vehicle (14).

10. An arrangement or method according to any preceding claim, characterised in that the closure (12) is opened to the initial extent by a spring mechanism (34).

11. An arrangement or method according to any preceding claim, characterised in that the initial opening means (34) is an electric actuator.

12. An arrangement or method according to any preceding claim, characterised in that the controlled opening mechanism (18) is a gas spring.

13. An arrangement or method according to any preceding claim, characterised in that the closure (12) is an openable motor vehicle door or lid.

14. An arrangement according to claim 13, characterised in that the door is a hatchback (12).

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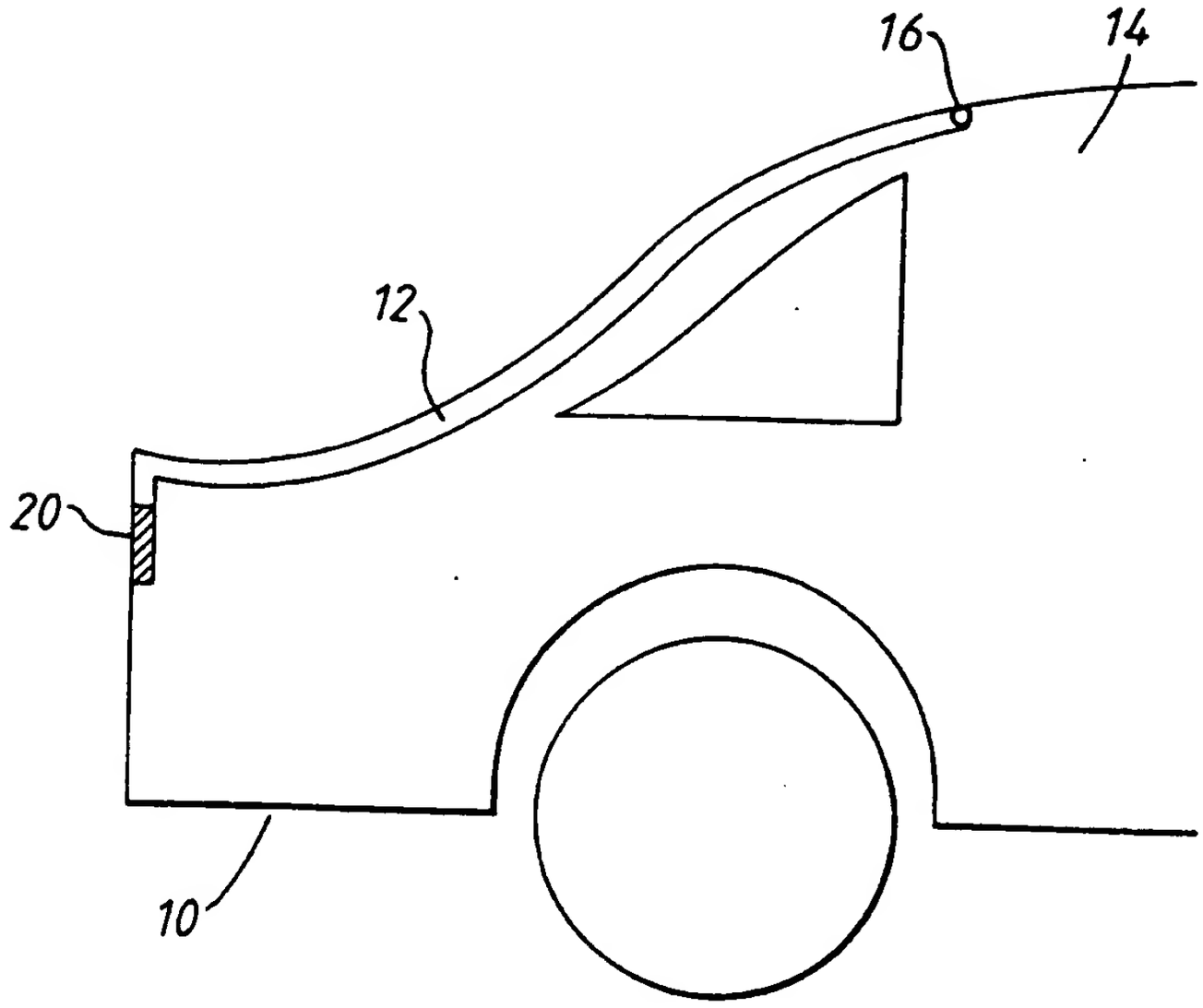

Fig. 1

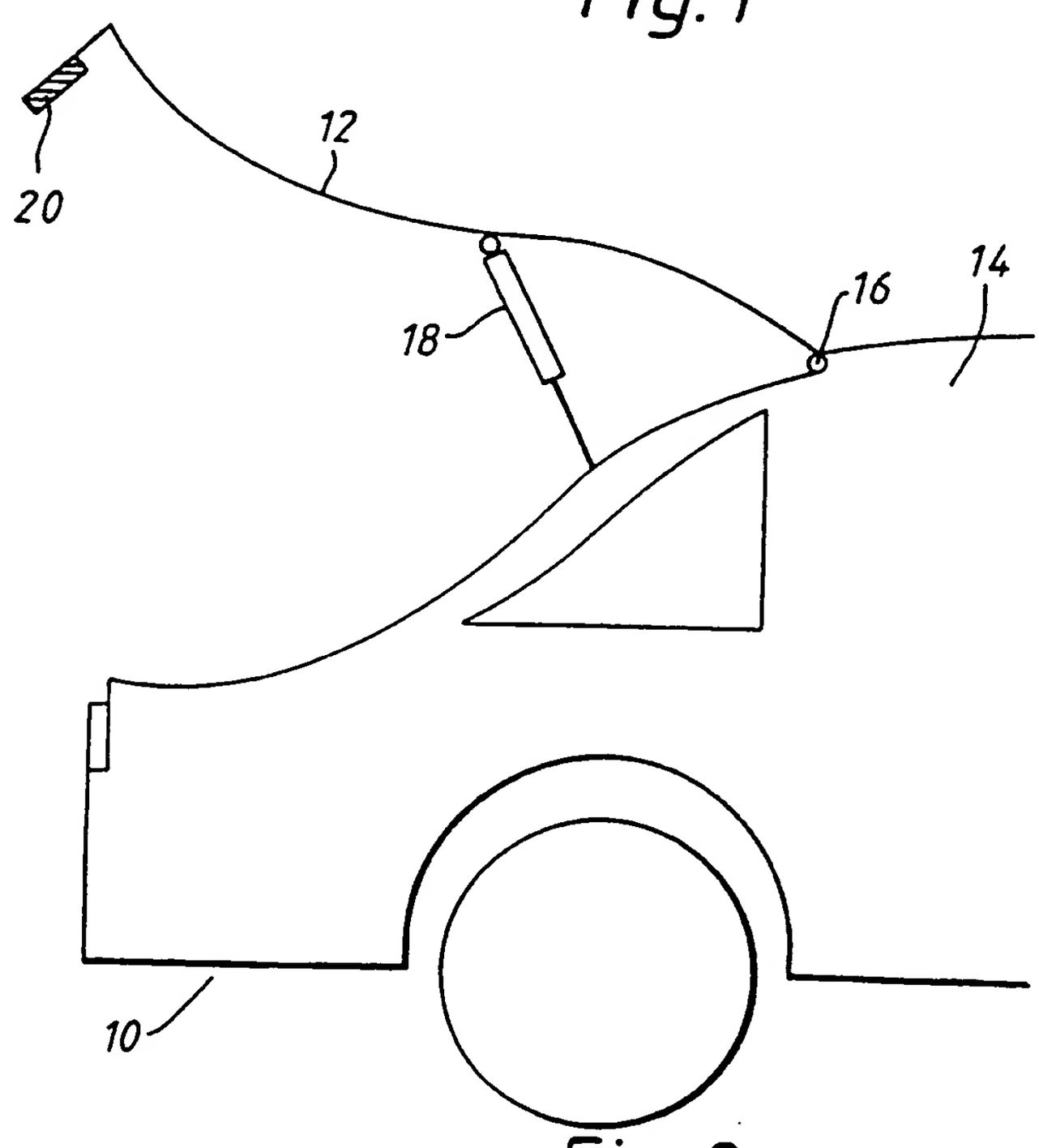

Fig. 2

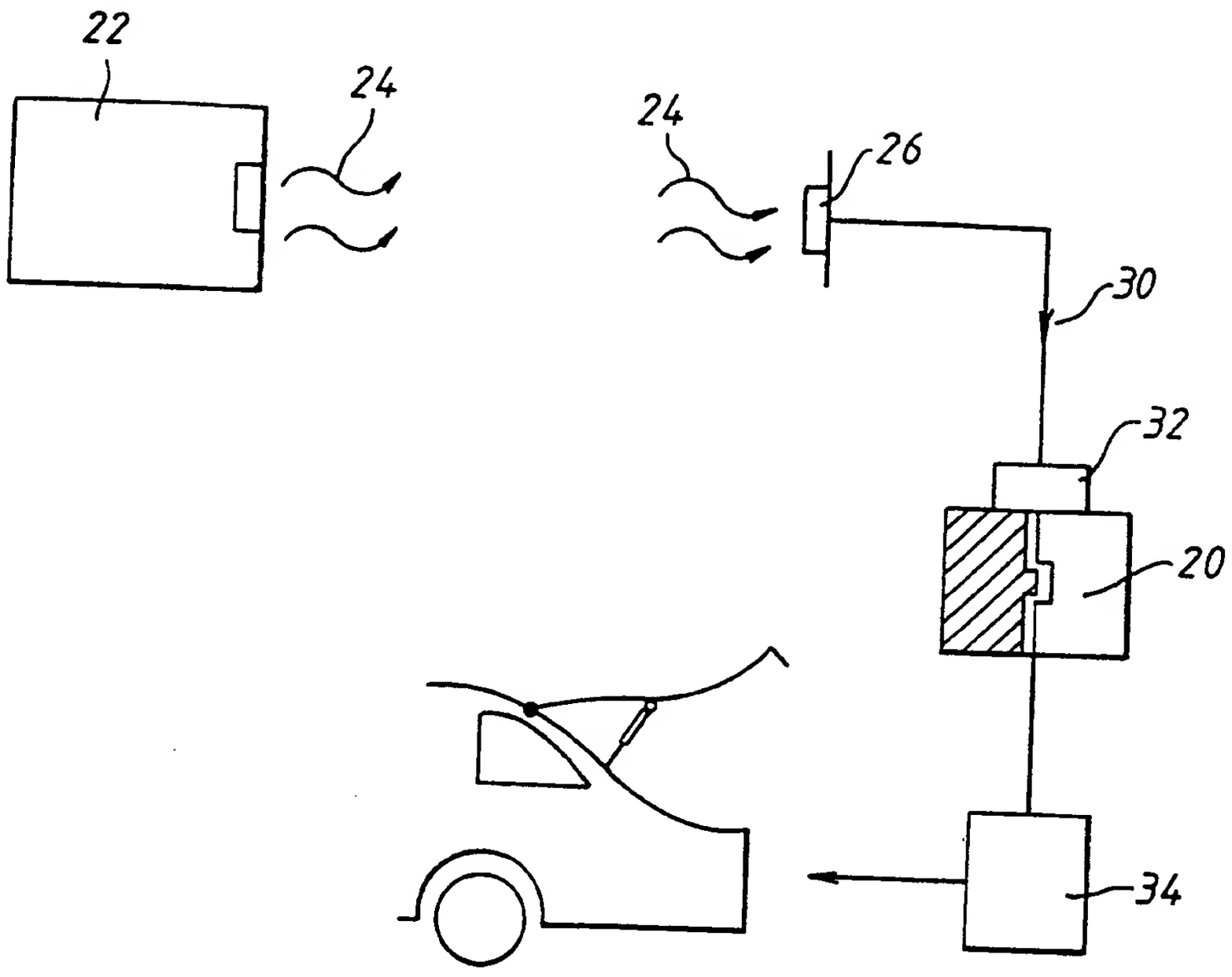

Fig. 3

INTERNATIONAL SEARCH REPORT

In' nal Application No
PCT/GB 96/01453

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 E05B65/19 E05F1/08

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 E05B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	US,A,4 739 585 (PICKLES) 26 April 1988 see the whole document ---	1,4,10, 11 2,3,5-9, 12
Y	WO,A,92 14018 (AUDI AG) 20 August 1992 see the whole document ---	2,3,5,6, 12
Y	DE,A,42 18 798 (MERCEDES BENZ AG) 9 December 1993 see the whole document ---	7-9
X	US,A,4 746 153 (COMPEU ET AL.) 24 May 1988 see the whole document ---	1,4,11

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

* Special categories of cited documents :

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
6 September 1996	19.09.96
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl, Fax (+ 31-70) 340-3016	**Authorized officer** Westin, K

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INTERNATIONAL SEARCH REPORT

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PCT/GB 96/01453

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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X	US,A,3 081 078 (LOHR) 12 March 1963 see the whole document ---	1,4,11
A	DE,A,43 21 586 (OHI SEISAHUSHO CO., LTD.) 5 January 1994 see the whole document -----	1,4,12

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INTERNATIONAL SEARCH REPORT

Information on patent family members

Ir ional Application No
PCT/GB 96/01453

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